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VACUUM LEAK DETECTION AS APPLIED TO MAJOR SPACE ENVIRONMENTAL CHAMBERS

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Aerospace Environmental Facility
ARO, Inc.

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a subsidiary of Sverdrup and Parcel, Inc.

August 1963
ARO Project No. SM2301

ABSTRACT

The role of leak detection in the successful operation of large-scale space simulation chambers is discussed and a general leak detection program outlined. Methods, techniques, and equipment capabilities are presented along with the results of an actual leak detection effort conducted on a 10,000-cu-ft space simulation chamber.

PUBLICATION REVIEW

This report has been reviewed and publication is approved.

William D. Clement
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AF Representative, AEF
DCS/Test

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Colonel, USAF
DCS/Test

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1.0 INTRODUCTION

The development of large-scale space simulation test equipment (Refs. 1 and 2) has caused the field of vacuum leak detection to become a matter of major concern to the builders and operators of such equipment. This is primarily due to the continuous requirement for leak detection imposed by the many configuration changes required in developmental testing and the effect of poor leak detection on the efficiency of an operation characterized by long pumpdown-cooldown times.

Several factors contribute to the difficulty in efficiently performing the leak detection mission. The most important of these, aside from the sheer size and complexity of the surfaces to be checked, is the inherent difficulty in separating the actual leakage from the virtual leakage and outgassing effects without waiting for the system to reach equilibrium. In the case of a major space simulation chamber (Figs. 1 and 2), this effort is complicated by the presence of multiple pressurized subsystems containing a variety of liquids and gases, a large number of chamber penetrations, and a test vehicle containing a variety of virtual leakage and outgassing sources.

In view of the conditions described above, it is seen that a considerable number of false starts and wasted pumping time will be experienced unless a systematic program exists for quickly isolating and quantitatively determining the magnitude of the leakage from every possible leak source in the entire system. Such a program must begin with the design of each component installed in the system and continue as an integrated part of the overall operation if a prompt and accurate appraisal of the situation is to be made when trouble occurs.

2.0 METHODS, TECHNIQUES, AND EQUIPMENT

A wide variety of methods, techniques, and equipment is available for use in leak detection. The advantages and disadvantages of those most commonly used are discussed with emphasis being placed on the capabilities and applications of the mass spectrometer residual gas analyzer and mass spectrometer leak detector.

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2.1 CHANGE OF INTERNAL PRESSURE IN SUSPECTED SYSTEM

The existence of a significant leak from a system exposed to the vacuum environment (such as the internal system shown in Fig. 3) may be verified by changing the internal pressure of the system when the vacuum chamber is in operation. The resulting change in the magnitude of the leak may be determined from the known pumping performance, with the mass spectrometer residual gas analyzer, or from the change in chamber rate-of-rise ($V\frac{dp}{dt}$) with the pump valved off.

2.2 SOUND

The sound created by a large leak may in some cases be used to determine the location of the leak. Commercial leak detectors which amplify the sound are available, but their application is usually limited to large leaks and areas of low background noise level.

2.3 SOAP FILM

A convenient method of locating leaks on the order of 10^{-2} atm cc/sec and larger is by applying a soap film to the external surface of a pressurized system. The leaking gas will create soap bubbles, the size being proportional to time and the magnitude of the leak. Practical application of this method is limited to the large leaks because of the difficulty of applying the film without creating bubbles and maintaining a continuous film, particularly on vertical surfaces.

2.4 DYE CHECK

Large leaks which are accessible from both sides may be located by the establishment of a pressure differential across the leak and application of a dye solution to the high pressure side. Given sufficient time, the dye will appear on the low pressure side of the leak.

2.5 TRACER GAS EFFECT ON PRESSURE GAGE

The characteristics of many vacuum gages are affected by the composition of the gas to which they are exposed. As an example, the ion current from an ionization gage is decreased by the presence of helium in the gas being measured. A leak can thus be detected by the application of helium to suspected areas until the ionization gage indicates the

presence of helium in the system. Liquid acetone may also be used as the tracer, but the effect is an increase in ion current from the gage. This method is particularly applicable to small vacuum systems.

2.6 HALOGEN LEAK DETECTOR

The fact that the positive ion emission from a heated platinum element is increased by the presence of a halogen compound is the basic principle of operation of the halogen leak detector. Commercial models are capable of sensitivities on the order of 10^{-5} atm cc/sec. The methods of application of the instrument to a vacuum system are essentially the same as with the mass spectrometer leak detector discussed in section 2.8 and illustrated in Figs. 7 and 8. A gas containing traces of a halogen compound is used as the tracer for leak detection.

2.7 MASS SPECTROMETER RESIDUAL GAS ANALYZER

The mass spectrometer residual gas analyzer (Ref. 3), as applied to leak detection, is used chiefly to identify the gases present in the system at any given time and to indicate their relative rates of increase by successive scans. This equipment plays a vital role in a well-balanced leak detection program and should therefore be regarded as basic equipment for any large space simulation system. In a system where hydrocarbons are likely to be present, it is desirable that it be capable of covering a mass range of from 2 to approximately 200 with unit resolution and that it simultaneously display several selected mass numbers.

The principal limitation to the use of the residual gas analyzer in leak detection is that it cannot be operated at pressures above 10^{-5} to 10^{-4} torr. In a system where severe leakage prevents the attainment of this pressure level, the analyzer must be used by throttling its inlet and supplying the desired operating pressure with an auxiliary pumping system. It is important to note, however, that this type of operation imposes the additional limitation that the selective pumping speeds of the various mixture components will cause an apparent change in the composition of the mixture. In this case, it is necessary to correct for this condition unless it is sufficient to note simple changes in the composition of the mixture without requiring a knowledge of the actual percentages present.

2.8 MASS SPECTROMETER LEAK DETECTOR

To date, the mass spectrometer leak detector has proven to be the most useful and versatile leak detection instrument; for this reason, its operating principle and methods of application are discussed in some detail.

The mass spectrometer leak detector is a specialized version of the mass spectrometer which is designed to detect one or more specific tracer gases. It can be used to accurately determine the location and magnitude of a wide range of leak sizes and can be operated over a wide range of pressures by the proper manipulation of the valving included.

The basic principle of operation and the various components of a typical detector are illustrated in Figs. 4 and 5, respectively. The gas sample, which is a mixture of different gases, enters the analyzer tube and passes through an electron beam where a number of the gas molecules are struck by the electrons, causing the loss of an electron from the molecule which results in a positive ion. The ions are then electrically accelerated and collimated into a magnetic field which causes the ions to follow a curved path, the radius of which is proportional to the ion mass-to-charge ratio (m/e), the velocity, the accelerating voltage, and inversely proportional to the magnetic field strength. The ions are thus separated according to their m/e values and the other variables adjusted such that only the ions of a certain gas will strike the collector plate. This gas is then used as the tracer gas for leak detection.

The ions striking the plate cause a current flow which is proportional to the partial pressure of the gas to which the mass spectrometer is tuned. The partial pressure of the tracer gas in the sample is directly proportional to the magnitude of the leak admitting the tracer gas to the system and to the total pressure in the analyzer tube, and inversely proportional to the magnitude of the total gas load.

$$PP_{Tracer} = KP_{Total} \frac{q_{Tracer}}{q_{Total}}$$

where K is a factor to account for the difference of pumping speed for different gases (usually not significant for leak detection purposes).

The sensitivity of the detector is determined by the smallest tracer gas partial pressure which will produce a detectable current flow. It can be seen from the above equation that the sensitivity of the instrument is increased by increasing the total pressure in the analyzer tube. The maximum operating pressure is usually between 1×10^{-4} and 2×10^{-4} torr because of short tube life at higher pressures. The ratio

of the tracer gas partial pressure to the total pressure is equal to the volumetric concentration of tracer gas in the sample:

$$\frac{PP_{Tracer}}{P_{Total}} = K\frac{V_{Tracer}}{V_{Total}} = K\frac{q_{Tracer}}{q_{Total}}$$

A calibration of the instrument with samples of known concentration will produce a leak detector meter reading vs tracer gas concentration curve similar to the one shown in Fig. 6. The calibration must be conducted at a constant total pressure in the analyzer tube. Two curves should be obtained: one at the normal tube operating pressure, and one that is a pressure decade lower to be used in applications where the higher pressure cannot be maintained.

Some mass spectrometer leak detectors are designed to be used with only one tracer gas, usually helium, whereas others are capable of detecting several gases, such as helium, argon, and neon. Instrument sensitivity is reduced in the combination design, but an attractive feature is the ability to change tracer gases when the background concentration of another is high.

The method of connecting the leak detector to the vacuum system is dictated by the system pressure and the detector sensitivity desired. The methods most commonly used are:

1. The detector is connected directly to the vacuum chamber as shown in Fig. 7. This method is applicable only at chamber pressures equal to or greater than the desired analyzer tube pressure. The leak detector throttle valve may be throttled to maintain the desired analyzer tube pressure.
2. The detector is connected to the foreline of the pumping system as shown in Fig. 8. This method is satisfactory for the entire range of chamber pressures because the forepressure is essentially constant.
3. The leech, a specialized method of attaching the detector to a surface that cannot otherwise be evacuated, is illustrated in Fig. 9. In principle, this method is essentially cases 1 or 2 above, depending on the pumping system used.

The tracer gas may be applied to the system in a wide variety of methods, those most commonly used are as follows:

1. With the leak detector connected to an evacuated system, the suspected area is "probed" with a fine jet of tracer gas or covered with a plastic bag (Fig. 10) whose edges are securely taped, and tracer gas injected into the bag. The bagging technique is preferred in cases where a large area must be searched for a small leak and because it ensures a flow of pure tracer into the leak. A leak under a large bag may be pinpointed by successively dividing the area of the bag into two parts and rechecking each half. Care should be taken to avoid being mislead by traces of gas escaping from a probe (or a leaking bag) which may be picked up by a leak far from the spot being checked.

2. The "sniffing" method of application is illustrated in Fig. 11 wherein the system or subsystem is pressurized with tracer gas and suspected areas sniffed with the leak detector. A continuous gas sample is drawn into the leak detector by its own pump through a sniffing probe which contains a throttle valve. This method is not applicable to large search areas because the sniffing probe must be moved slowly and pass very near a small leak to cause an indication on the leak detector. Another disadvantage of this method is that most systems will retain large quantities of the tracer gas for long periods of time. The resulting high tracer background will delay the subsequent use of the bagging method until the background is reduced or a different tracer gas is used.

The successful application of the mass spectrometer leak detector is particularly dependent on a knowledge of the relationship between the system and instrument. In particular, the system response time and system sensitivity must be known before detailed leak checking is initiated. The response time of a system is defined as the time lapse between the application of tracer gas to a leak and the resulting indication on the leak detector. In large complex piping systems this may be several minutes. In systems with long response times, for instance, the point of application of tracer gas may have moved a considerable distance from the actual leak before the indication is noted on the leak detector. This can result in an erroneous conclusion as to the location of the leak unless the response time is known.

The system response time and sensitivity may be determined in one operation. When the system is being prepared for detailed leak checking, a standard leak of known size is installed at the farthest point in the system from the leak detector. When tracer gas is admitted to the system through this leak, a response time sensitivity curve similar to Fig. 12 will result. The standard leak used should be the smallest leak that will produce a readable signal in a reasonable length of time. Then when detailed leak checking begins, a waiting period at least equal to the maximum response time should be allowed between application of the tracer gas to different areas. Occasional re-calibration with the standard leak may be necessary if the system is such that the background gas load changes over a period of time.

3.0 DISCUSSION OF A TYPICAL LEAK DETECTION PROGRAM

In view of the expense and time involved in the operation of a large system, it is necessary that a thorough engineering analysis of the situation be carried out before a system shutdown or the initiation of any detailed leak checking efforts. The basic questions which must be answered are:

1. Does a leakage problem actually exist?
2. What is the total leakage rate of the system and how is it distributed between the chamber itself and the various internal subsystems?
3. Once the leakage is traced to a given subsystem and evaluated, what are the conditions under which it occurs and what methods, procedures, equipment, and special conditions are required for its detection?
4. When does the effect of repairs achieved justify an attempt to put the system in operation?

If these questions are answered on a sound engineering basis, there is little danger of being mislead by an apparent leak, such as that caused by the thermal overloading of a cryosurface (Ref. 4), or expending effort in repairing a leak that is not actually affecting the system performance. In order to illustrate the use of the equipment and procedures discussed thus far, a typical leak detection program is outlined in the following sections.

3.1 IDENTIFICATION OF GASES MAKING UP TOTAL GAS FLOW TO PUMPS

The prime method of making this determination is the periodic scanning of the chamber contents with a mass spectrometer residual gas analyzer. The unscheduled presence of some gas, or the rates of increase of gases as indicated by successive scans, provides the first clue as to the nature and possible location of the leak or leaks. One of the simplest determinations of this type is the noting of an increase in oxygen along with nitrogen which indicates an air leak rather than leakage from a pure nitrogen source such as the various liquid nitrogen systems or bottled nitrogen gas aboard the test article. If, however, the increasing medium could have come from several sources, then each of these sources must be checked by the use of tracer gases which could only have come from that single source. If carefully performed, this procedure can provide positive evidence as to whether the systems checked are leaking into the chamber, but it should be noted that since these systems will contain gases other than the tracer, no quantitative measure of the leak rate will be produced.

3.2 ESTABLISHMENT OF SUBSYSTEM LEAK RATES

If the preceding analysis indicates that the total leak is emanating from a single subsystem, and since such a leak would be an appreciable percentage of the system pumping speed, an approximation of its magnitude can be obtained by changing the pressure in the leaking subsystem by a known amount and observing the effect on the system pressure.

If, however, the overall leak rate consists of several leaks, this procedure is not feasible. The required procedure in this case is to withdraw the liquid nitrogen, etc., from a system known to be leaking and pressurize this system with a tracer gas. The mass spectrometer residual gas analyzer or mass spectrometer leak detector can then be used to compare the signal received from this operation with that received from the injection of a calibrated leak of known size. In this way the actual value of the leak from this subsystem can then be calculated. By completing this process for all subsystems which can conveniently be checked by this method, the total system leak rate and its distribution can usually be established. Unless, however, the total leak rate established by the above method is seen to be capable of causing the pumping problem when compared with the system pumping performance curves, it may be necessary to accurately assess the main chamber leak rate by carrying out the ponderous procedure of enclosing the entire chamber in a tracer filled bag and comparing the resulting gas analyzer reading with the reading generated by a calibrated leak of the same tracer gas.

3.3 LOCATION OF LEAKS WITHIN THE INDIVIDUAL SUBSYSTEMS BEFORE THE SYSTEM IS SHUT DOWN

Although the procedures outlined in sections 3.1 and 3.2 definitely verify the presence of an actual leak in any given subsystem and will quantitatively establish the magnitude of the total leak in each such system, they can do little toward locating the leak within the system or defining whether one or more leaks exist. It is at this point that a properly designed system can materially shorten the process and in some cases allow the test to be continued by isolating the leaking portion of the system. The prime means of achieving this attractive objective is the compartmentation of such large systems as the helium cryosurfaces and associated liquid nitrogen shielding. Such inbuilt compartmentation together with pumpout ports, tracer inbleed ports, and drainage systems is particularly desirable in the case of very large systems since it may otherwise have to be performed by partial disassembly of the system during leak check in order to facilitate decontamination of the systems and to reduce the system response time to a reasonable value.

3.4 DETAILED LEAK CHECKING OF INDIVIDUAL SUBSYSTEMS OR COMPARTMENTS OF SUCH SUBSYSTEMS AFTER THE SYSTEM IS SHUT DOWN

It is at this stage that the very large expenditures of time and effort can take place, and it is for this reason that the steps given in sections 3.1, 3.2, and 3.3 must be accurately performed before such detailed work is begun. This is readily understandable when it is seen that the liquid nitrogen paneling in the system indicated in Fig. 2 contains approximately 18,000 square feet of surface and several thousand fittings and joints. Although the mass spectrometer leak detector is the mainstay in the detailed leak checking and is in all cases used in the final stages, a wide variety of methods, procedures and equipment can be used. The selection of the proper procedure is largely determined by the size of the leak under consideration although such considerations as contamination and physical location in large systems play a large part in this selection.

4.0 DESCRIPTION OF A MAJOR LEAK DETECTION EFFORT CARRIED OUT ON A LARGE SPACE SIMULATION CHAMBER USED FOR ROCKET PROPULSION TESTING

A schematic of the chamber is shown in Fig. 13, which consists of an 18-ft-diameter by 32-ft-long, liquid-nitrogen-cooled vacuum chamber

enclosed in an annular volume held at 10 torr during testing. The chamber, or liner, is constructed of stainless steel thermopanel and contains a variety of pressurized subsystems. Vacuum pumping is provided by a 1-kw, 20°K-helium cryopump, two 20-in. oil diffusion pumps, and two 5000-cfm roots blowers - mechanical pump combinations.

The system has been in operation for approximately two years (Ref. 5), and although leaks have occurred which required that the chamber be shut down with resultant losses in test time, the compartmentation features of the system have usually allowed the leaking subsystem to be quickly isolated. The leak sources inherent to this system are (see Fig. 13):

1. From the annulus
2. From liner LN_2 passages
3. From boundary-layer LN_2 passages
4. From the ejector LN_2 passages
5. From the internal LN_2 system
6. From the helium cryosystem
7. From the atmosphere through any feedthrough
8. From the atmosphere through the diffuser
9. From the atmosphere through the vacuum pump lines
10. From the vehicle fuel and supply lines
11. From the various test support equipment, such as cameras.

The leak detection effort being described was brought about by the appearance of a severe leak of approximately 100 atm cc/sec which first appeared at a liner temperature of 240°K and grew progressively worse as the chamber was cooled until the above figure was reached. Subsequent manipulations of the pressures within the various subsystems failed to have any effect on the system leak rate. The system was then warmed to about 240°K at which point the leakage was reduced to such a point that it could not be detected by the rate-of-rise method.

The individual systems were then cooled individually with no effect until the main liner temperature was reduced to 240°K. At this point, it was decided to shut the system down and submit the main liner liquid nitrogen system to a comprehensive leak check even though the pressure in the large system could not conveniently be changed to prove the presence of the leak.

This step was taken for the following reasons:

1. The leak was shown to be affected by the liner temperature.
2. All internal pressurized subsystems could be conclusively eliminated.
3. A leak from the atmosphere seemed unlikely since the chamber is almost completely enclosed by the annulus in which pressure changes produced no effect on leak rate.
4. The overall size and complexity of the system made "bagging" of the overall system impractical.
5. A reasonably thorough check of the large number of feedthroughs failed to disclose the leak.

The detailed leak check was begun by blanking off all valves and exhaust stacks, connecting a mass spectrometer leak detector to the system, and evacuating the liner nitrogen passages and associated piping. Since the accumulated moisture in the passages made the attainment of low pressures difficult, the system response time/sensitivity calibration was conducted at a system pressure of approximately 300 microns Hg. The injection of a 1.1×10^{-4} atm cc/sec helium leak at various stations produced the response times plotted in Fig. 14. (It is interesting to note that these times varied from 30 sec for the most distant station to approximately 15 min. for other locations. Although this effect was obviously caused by a viscous flow stream from the most distant point to the leak detector, whereas the flow from other locations was by diffusion only, it serves to point out one of the many pitfalls inherent to this type of investigation). Using the maximum response time of 15 min, the interior of the liner was then flooded with a 20-percent concentration of helium which, when compared with the standard leak, indicated a total leak rate of 2.2×10^{-4} atm cc/sec as indicated in Fig. 15. Although it was not thought likely that the chilling of a leak of this size could result in an expansion to 100 atm cc/sec, the bagging operation was completed with the result that three leaks were found whose total was in good agreement with the 2.2×10^{-4} atm cc/sec indicated by the overall check.

The chamber was again pumped down and cooled at which time the leak again appeared unchanged at 240°K. At this time, a comprehensive check of all chamber lead-throughs disclosed that the leak was in a remote instrumentation lead-through which was being affected by a physical movement caused by the thermal contraction of the liner as it was cooled.

Although the detailed leak checking of the liner contributed indirectly to the final isolation of the leak by accurately defining the size of the total warm leak in the liner, its net effect was the reduction of the chamber base pressure from the 10^{-6} to the 10^{-7} torr range. It can be seen from the preceding discussions that the use of a mass spectrometer residual gas analyzer would have quickly shown the leak to be an air leak and thereby would have saved valuable test time.

5.0 CONCLUSIONS

A well-organized and equipped leak detection effort can make a substantial contribution to the overall operational efficiency of a major space simulation system. Such an effort should be based on the quantitative use of mass spectrometer and associated equipment. Specifically, this equipment should include the following:

1. An on-line mass spectrometer residual gas analyzer.
2. Standard mass spectrometer helium leak detectors capable of extended use in the field with an instrument sensitivity of approximately 10^{-9} atm cc/sec.
3. Equipment capable of accurately determining the percentage of helium under a hood or bag.
4. A special mass spectrometer leak detector capable of using helium, neon, or argon for use on systems having a high helium background.
5. Various mechanical pumps, pressure gages, standard leaks, and the lesser forms of leak detection equipment such as halogen detectors, acoustic detectors, etc., for eliminating the larger leaks before the use of the mass spectrometer equipment.

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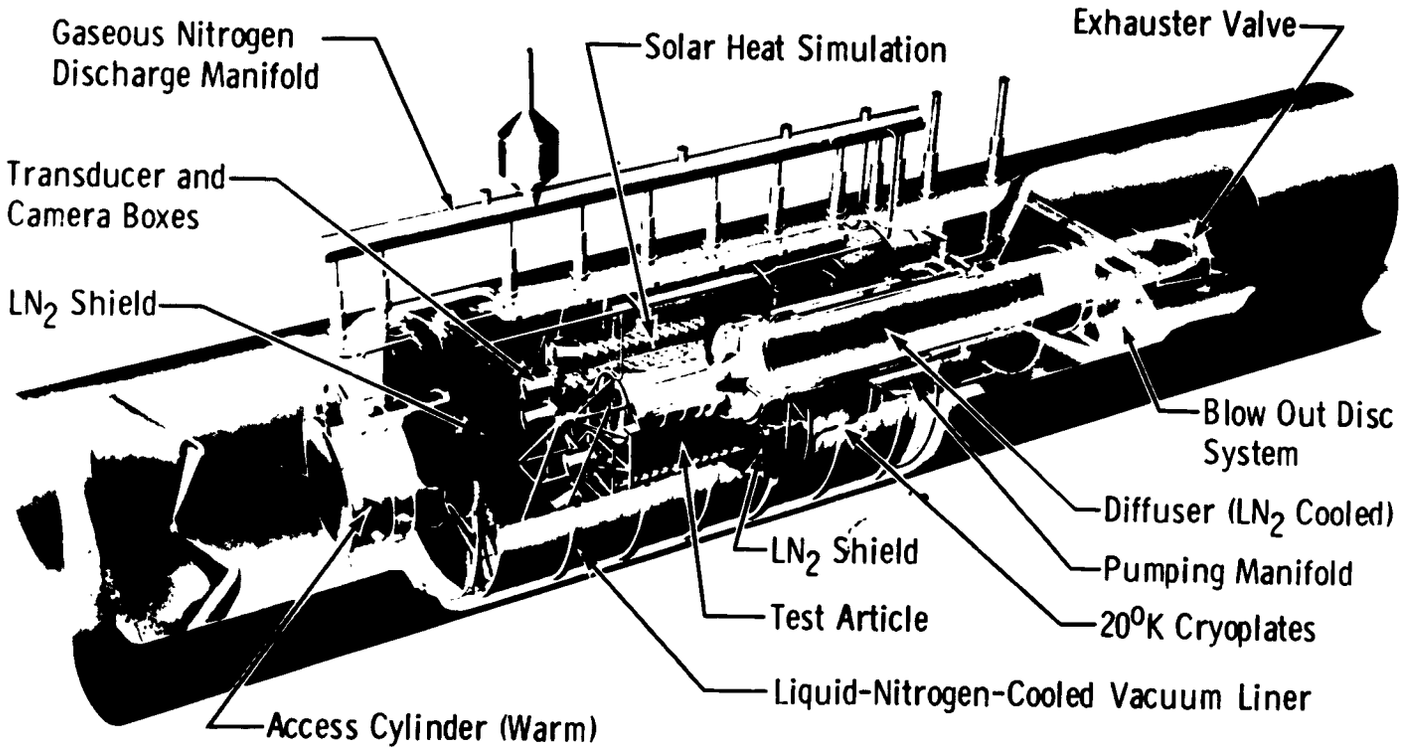

Fig. 1 Ultrahigh Altitude Rocket Cell J-2A

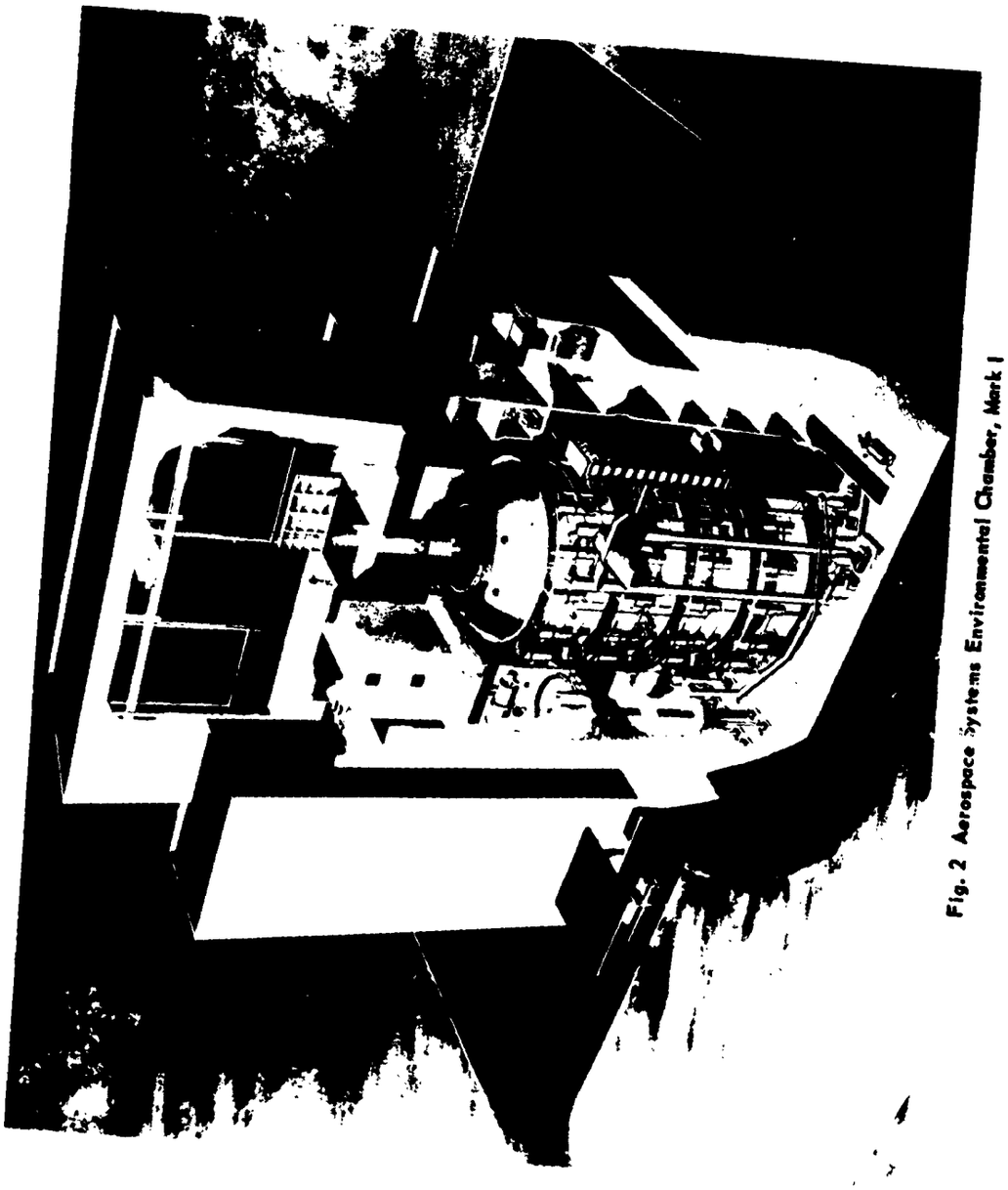

Fig. 2 Aerospace Systems Environmental Chamber, Mark I

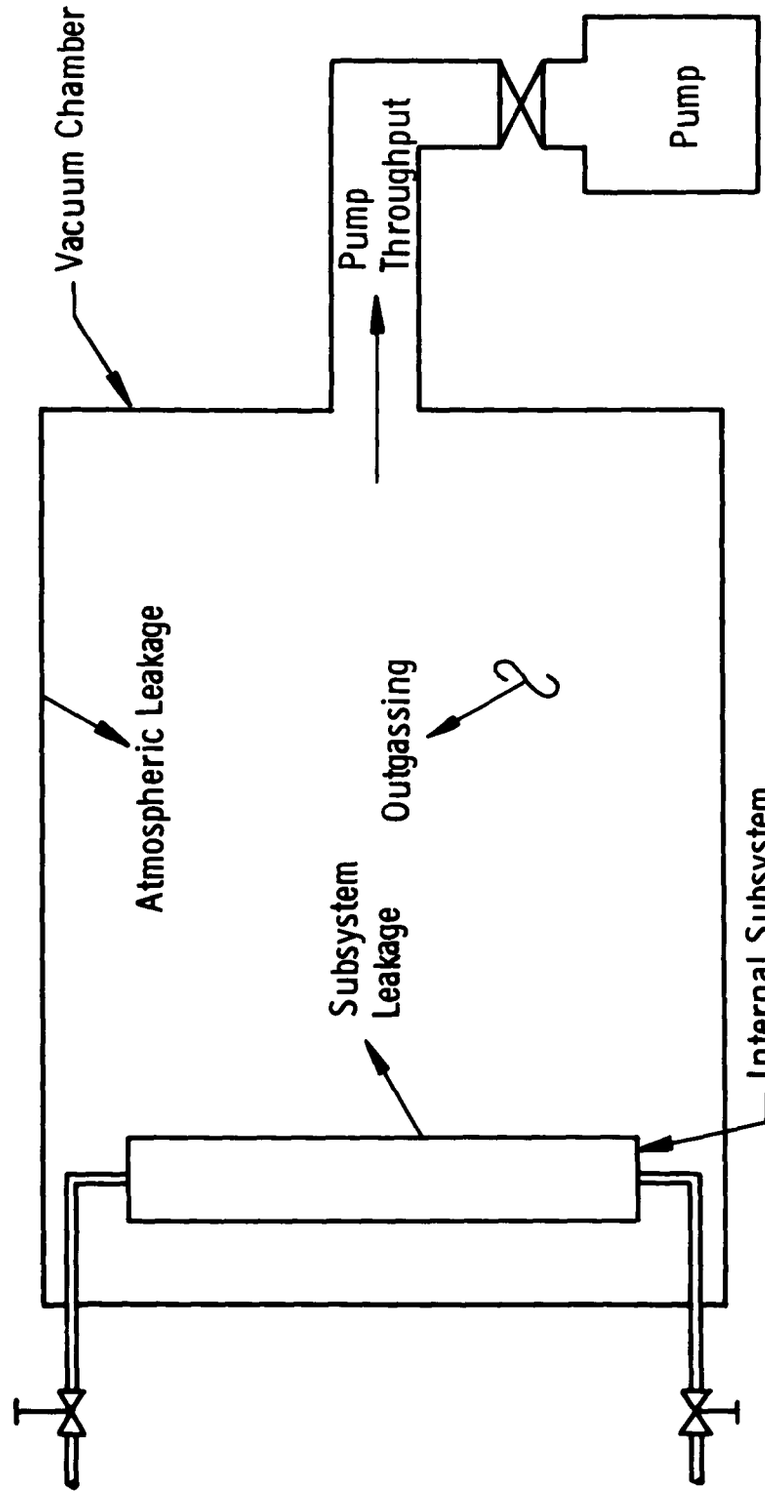

Fig. 3 Gas Sources in a Simple Vacuum System

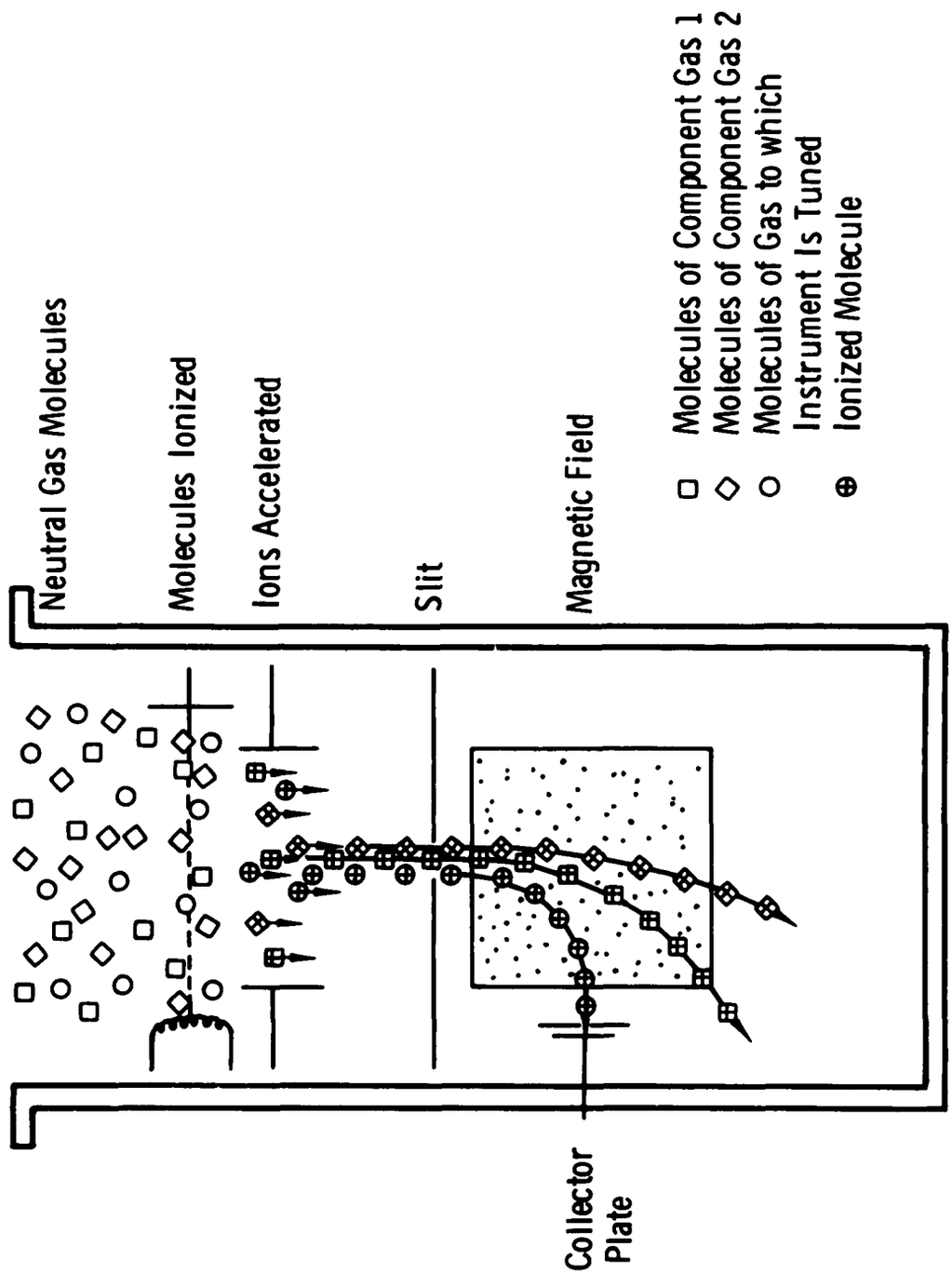

Fig. 4 Schematic of Analyzer Tube

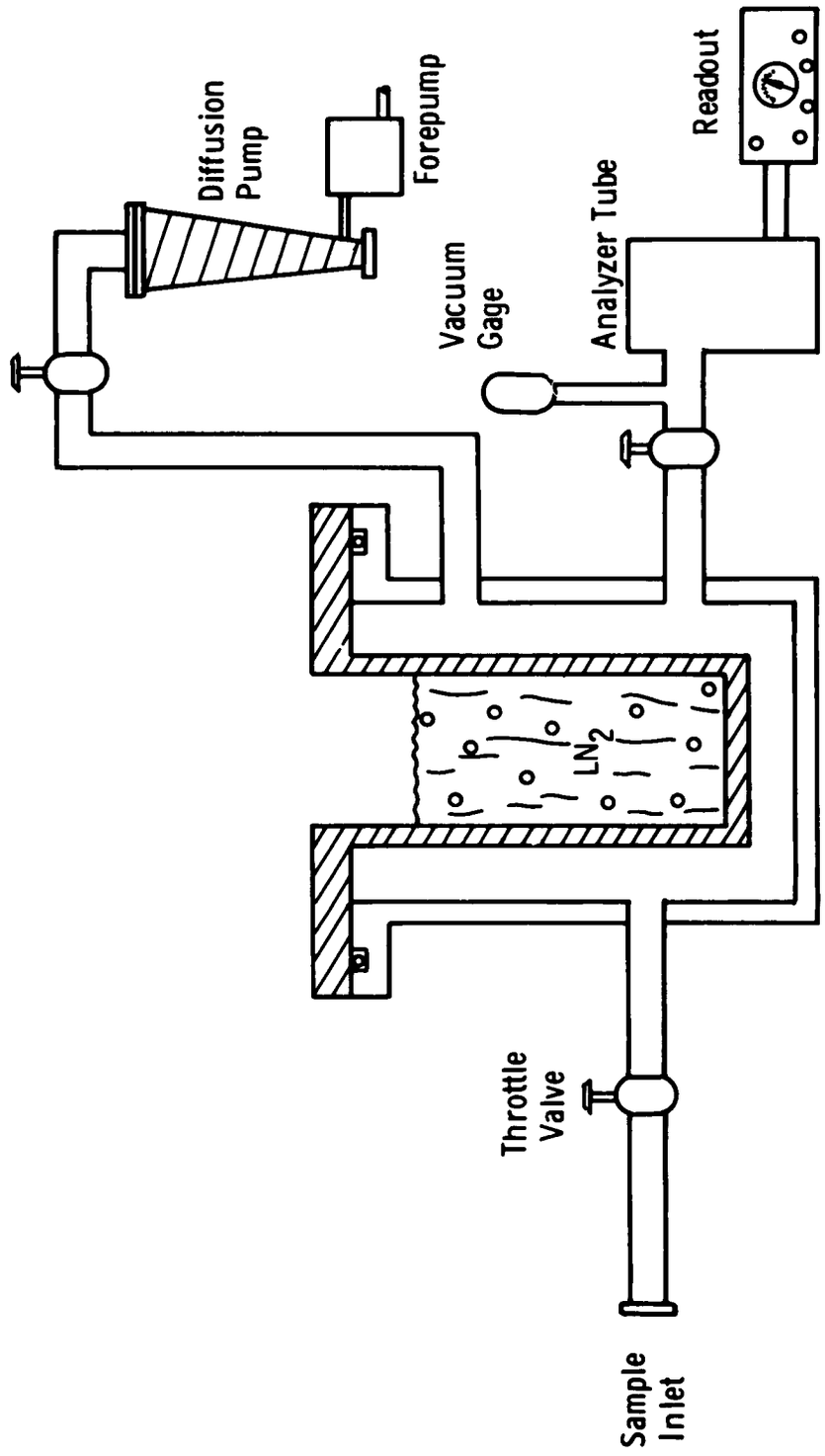

Fig. 5 Schematic of Leak Detector

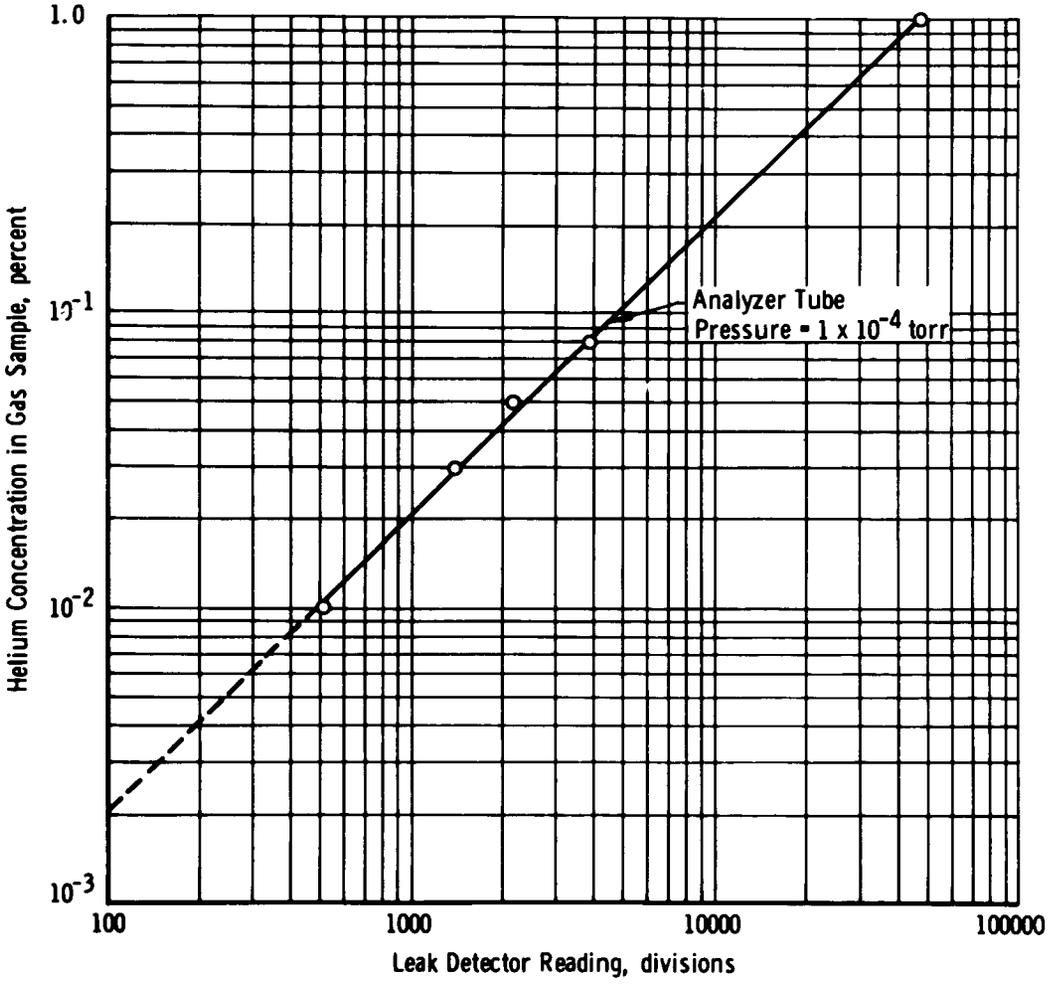

Fig. 6 Leak Detector Calibration

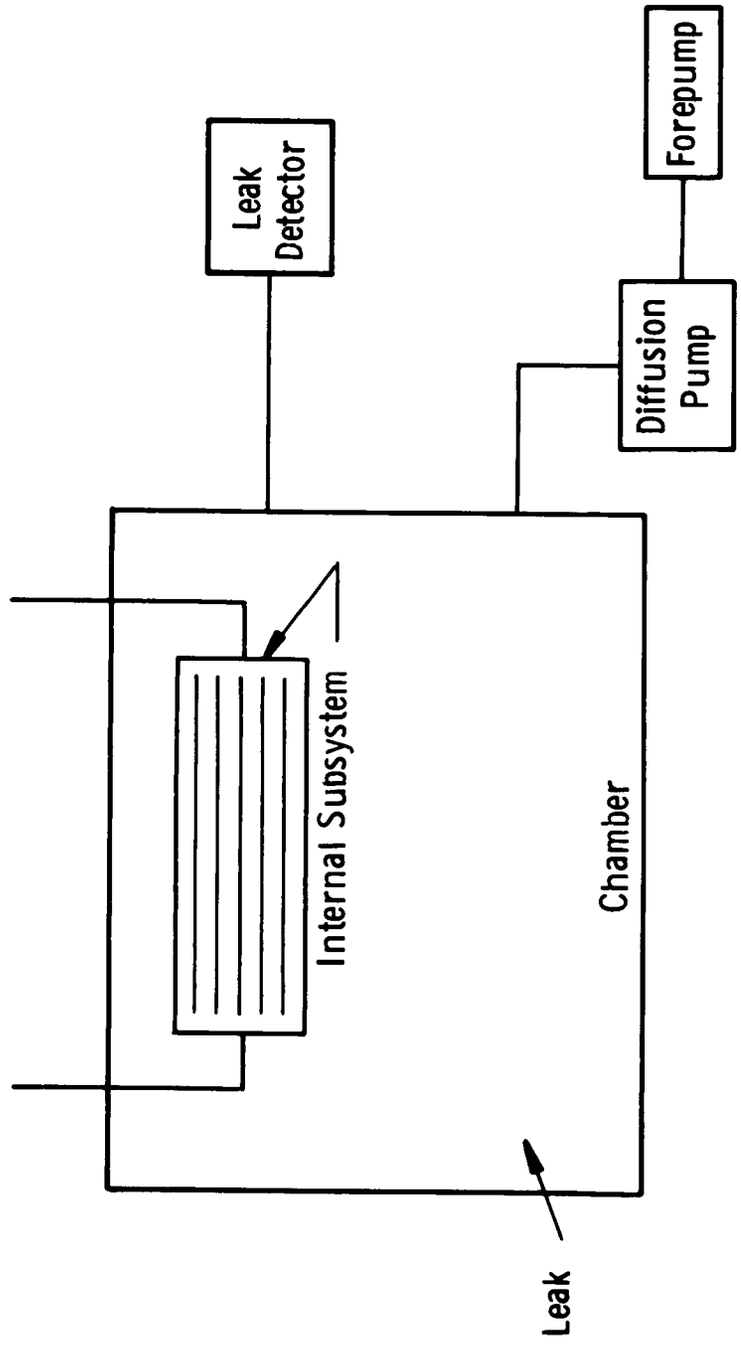

Fig. 7 Detector Applied Directly to Chamber

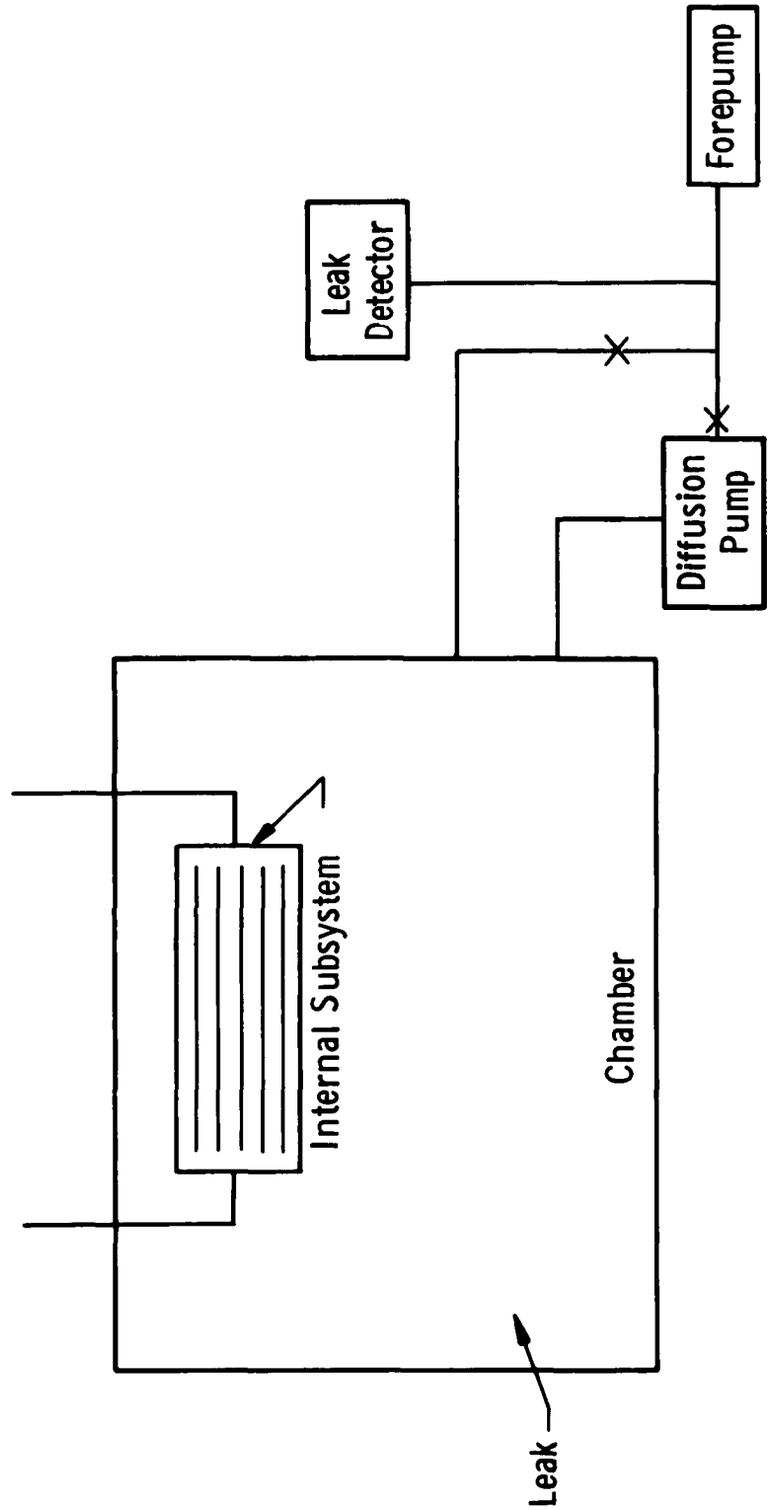

Fig. 8 Detector Applied to Forepump Line

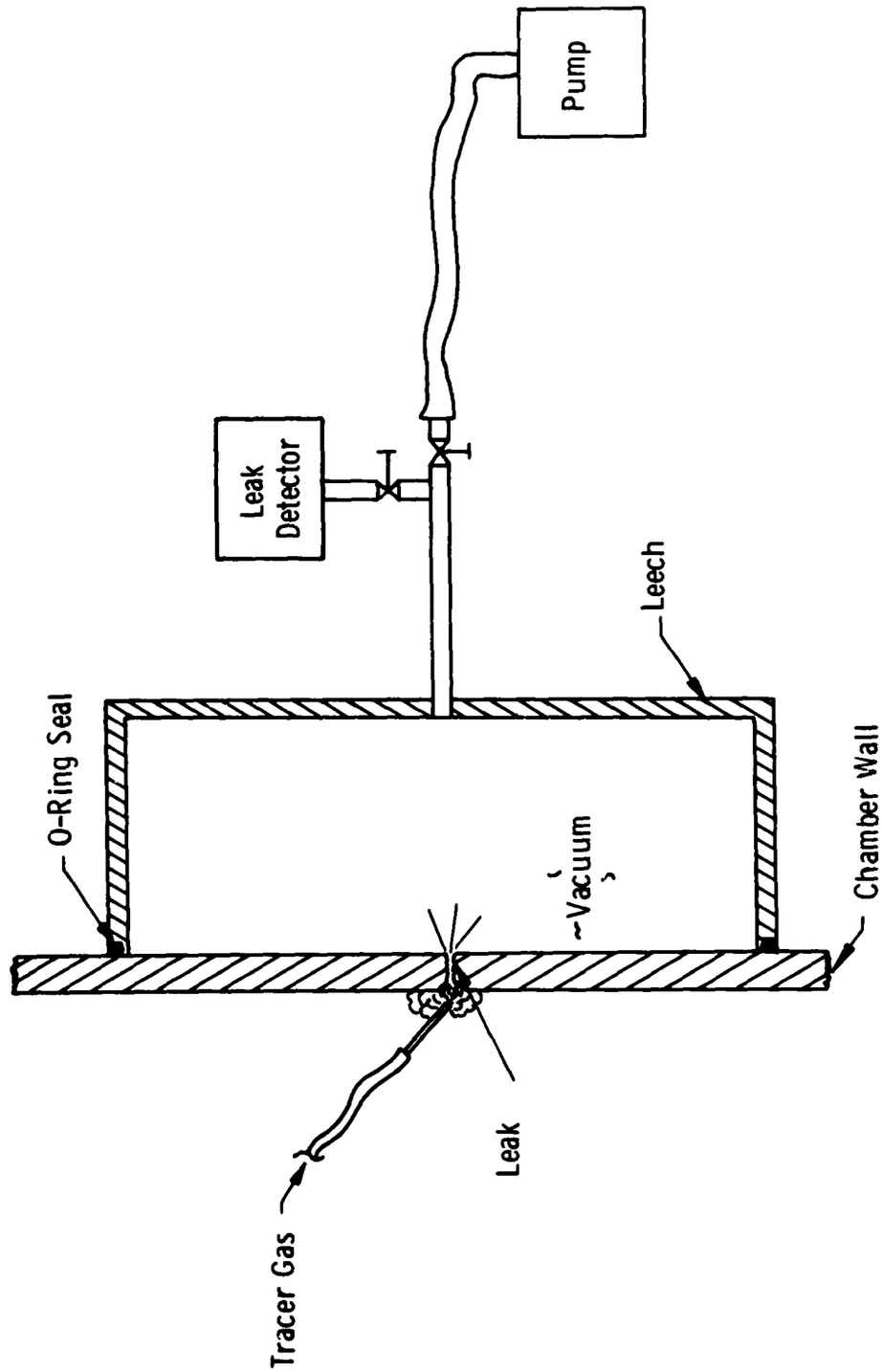

Fig. 9 Evacuation of Leakage Area by Leech Method

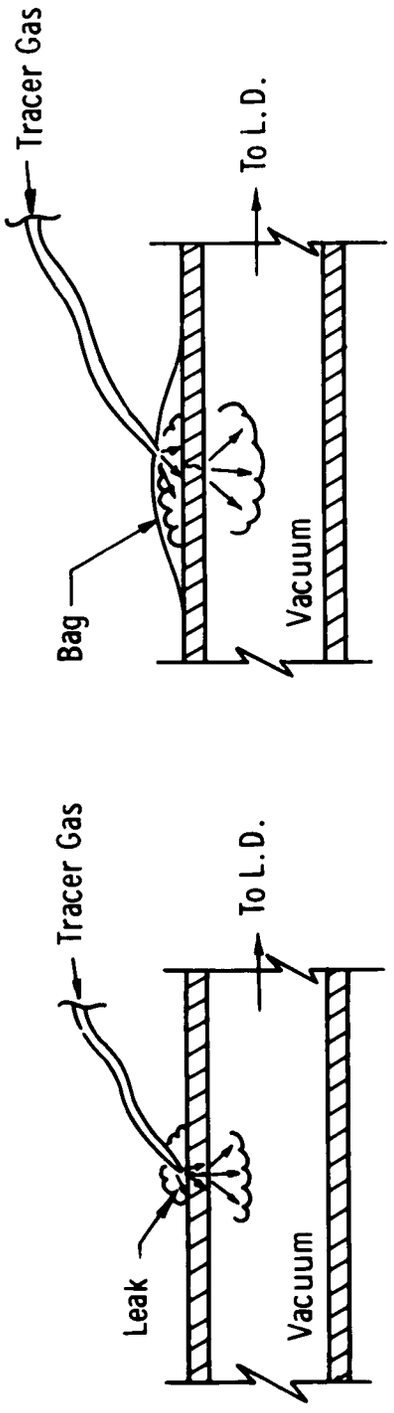

Fig. 10 Methods of Applying Tracer Gases to Evacuated Systems

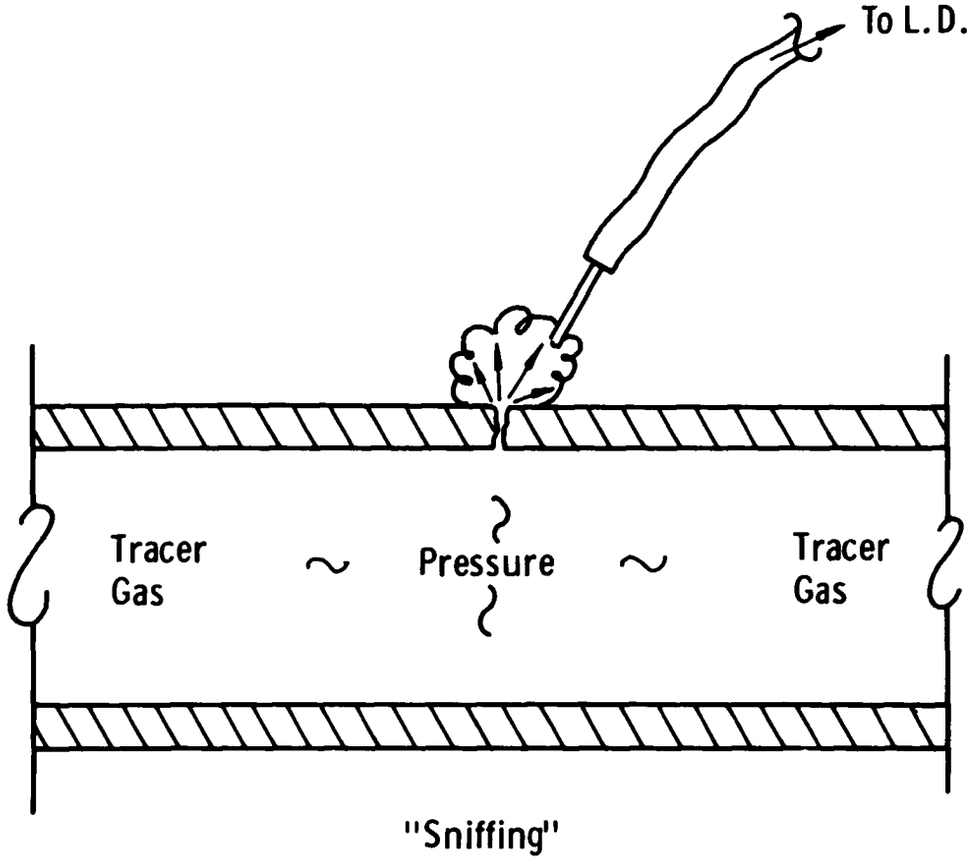

Fig. 11 Method of Detecting Tracer Gas in Pressurized Systems

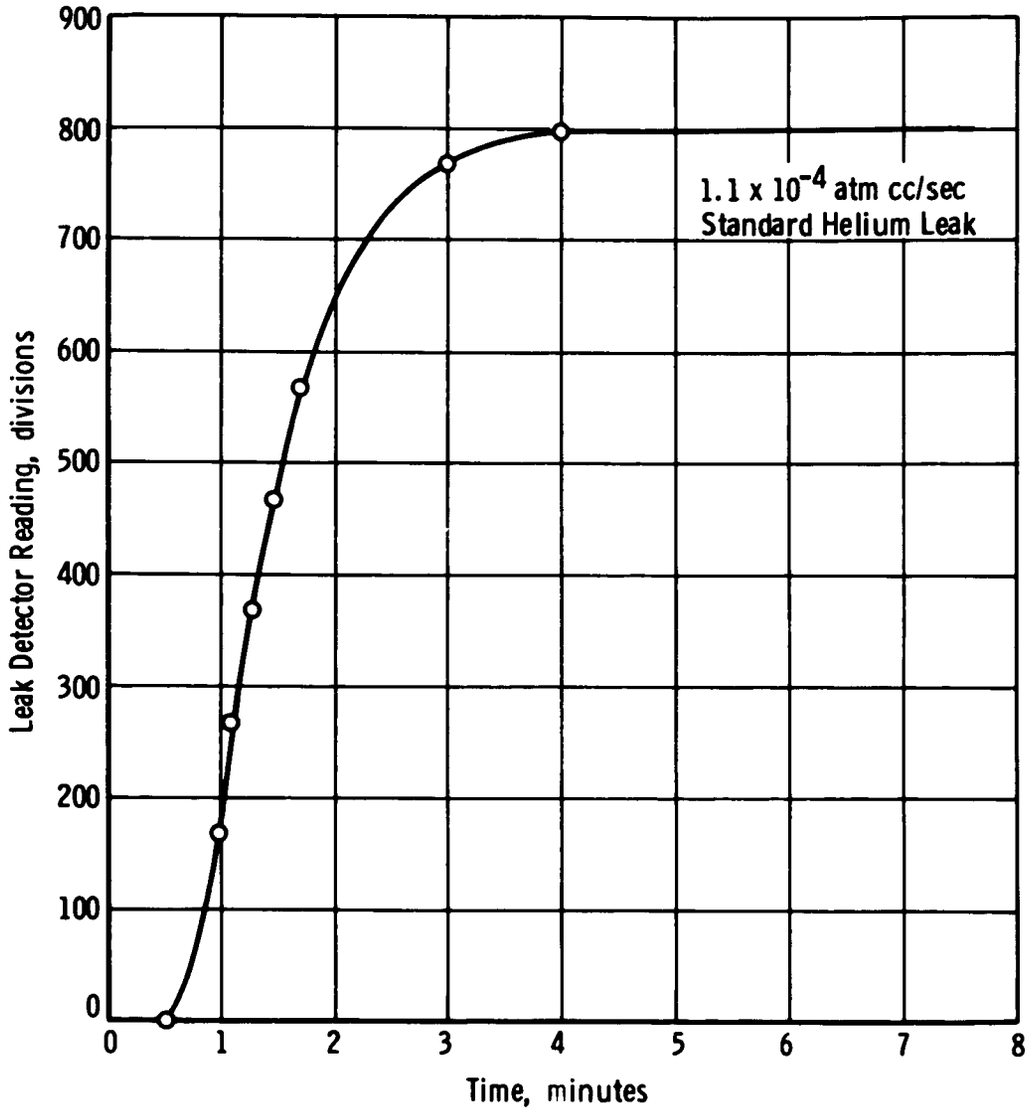

Fig. 12 System Sensitivity Calibration

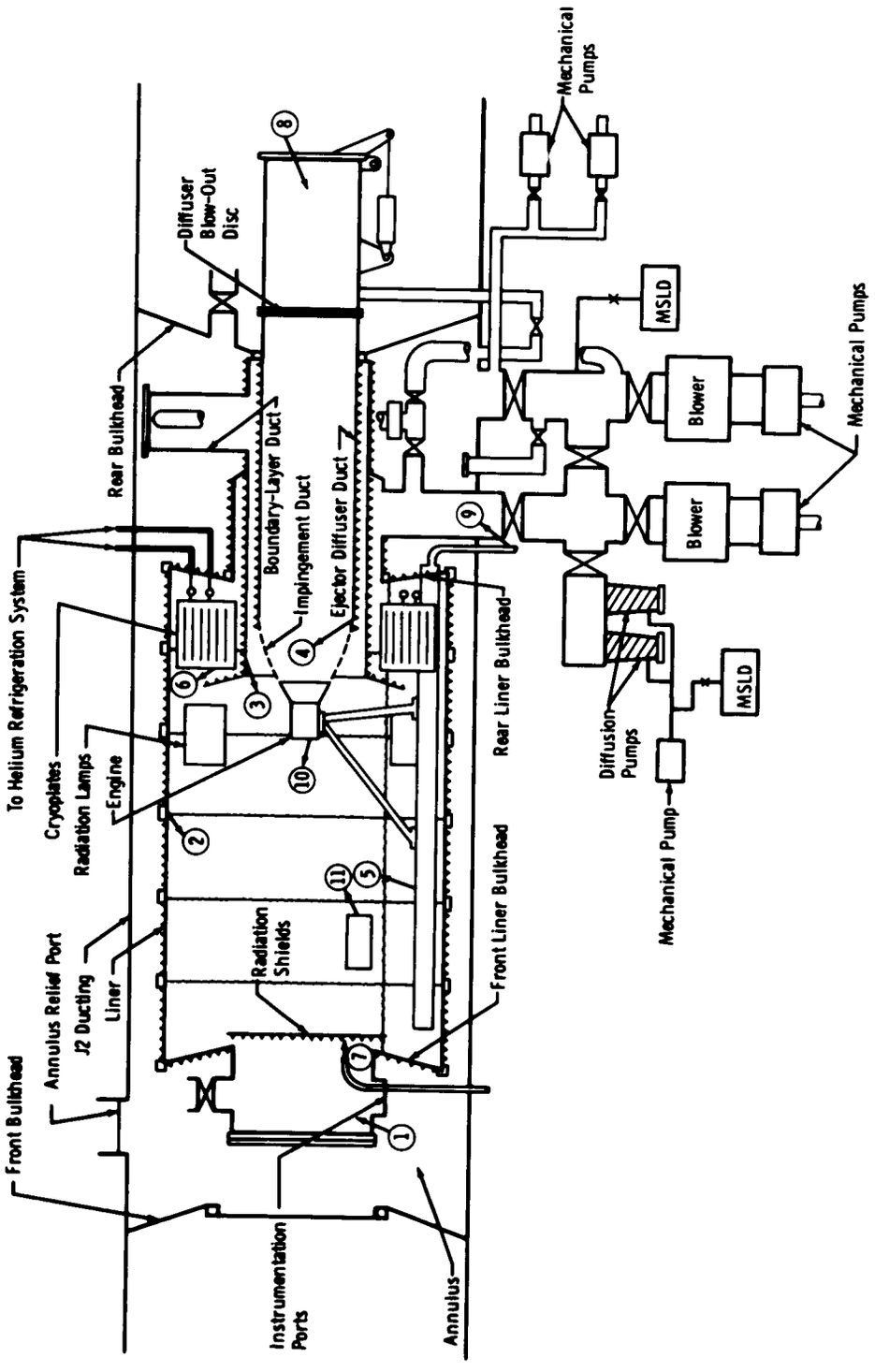

Fig. 13 Schematic of Ultrahigh Altitude Rocket Cell J-2A

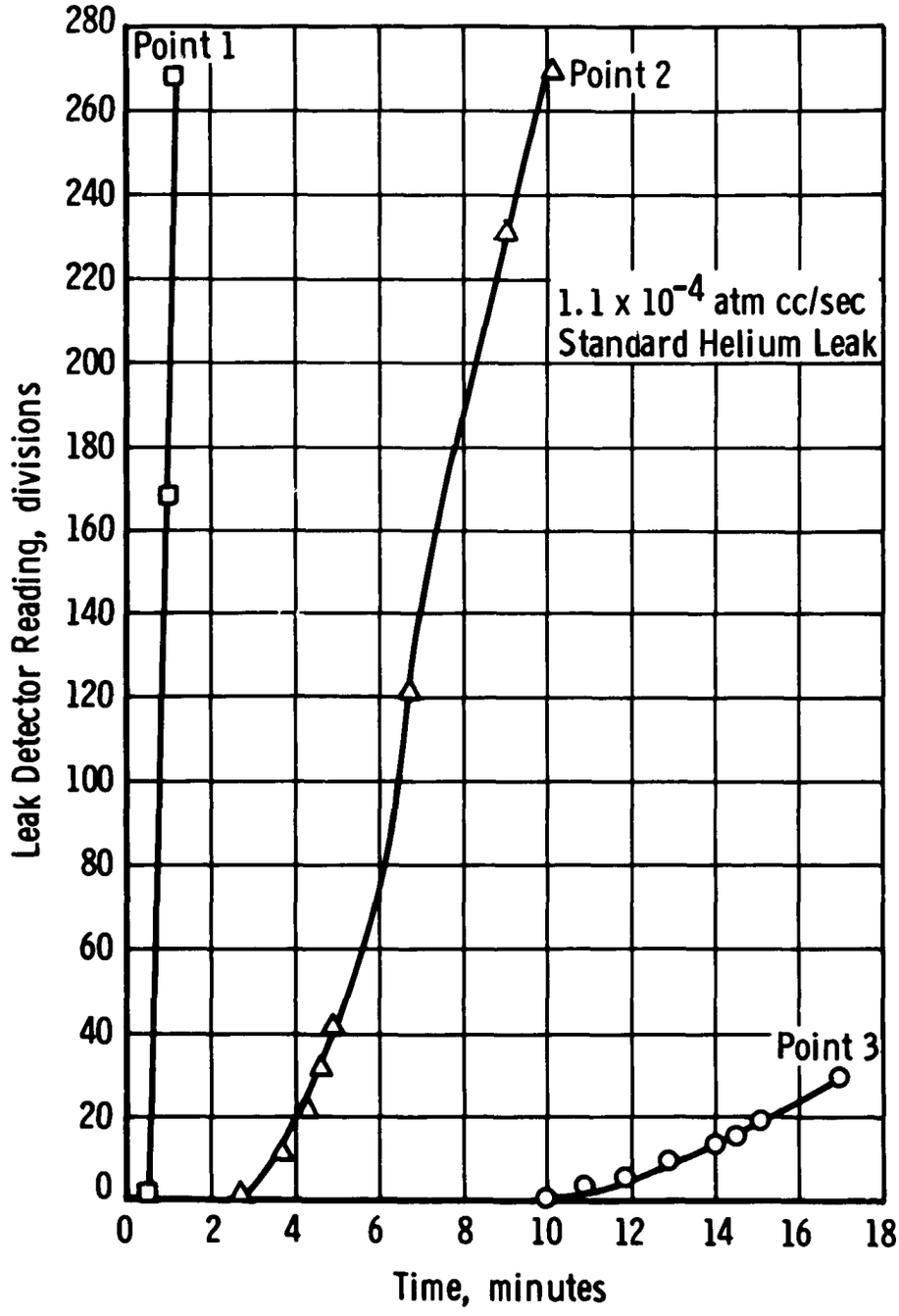

Fig. 14 System Response Time

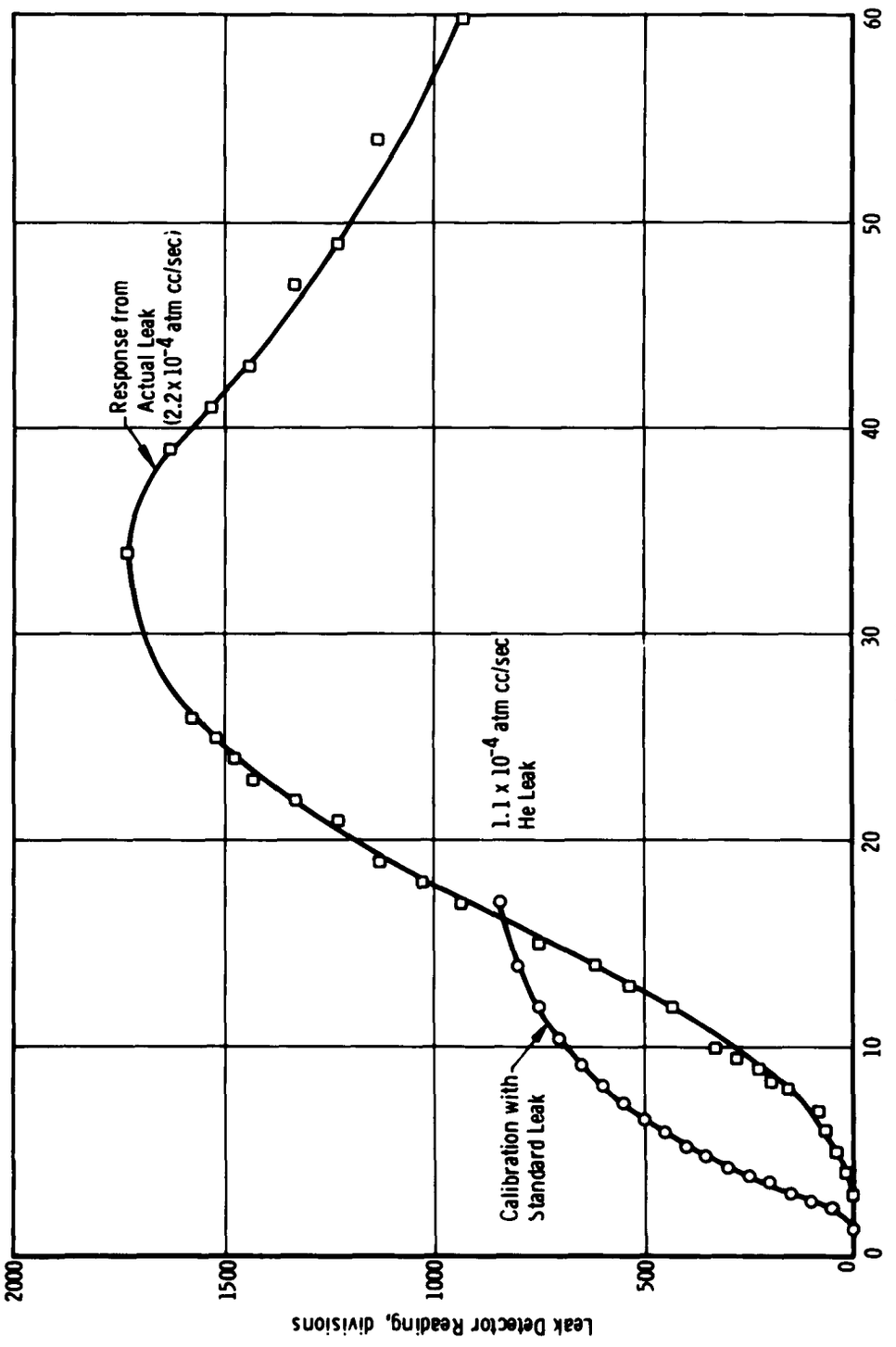

Fig. 15 Determination of Leak Size